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REPAIR PROCESSING METHOD AND WATER-REPELLENT TREATMENT METHOD OF WATER REPELLENT FILM

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REPAIR PROCESSING METHOD AND WATER-REPELLENT TREATMENT METHOD OF WATER REPELLENT FILM

[*Hassuisei himakuno ripea shorihouhoh, oyobi hassui shorihouhoh*]

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[*There are no amendments to this patent.*]

[*Translator's note: Names of products and companies are spelled phonetically in this translation.*]

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Specification

(54) [Title of the Invention]

Repair processing method and water-repellent treatment method of water repellent film

(57) [Abstract]

[Purpose] The purpose of the present invention is to provide a treatment method with improved bonding between the glass surface and a water repellent film in a repair treatment method for water repellent films used for automobiles.

[Means of solution] A repair treatment method for a water repellent film having a substrate layer characterized by the fact that the treatment includes (1) a process in which a plasma jet is applied to the aforementioned water repellent film to dissipate the water repellency and (2) a process where a water repellent solution is applied to the area without the water repellent function.

[Claims of the invention]

[Claim 1] A repair treatment method for water repellent films having a substrate layer characterized by the fact that the treatment includes (1) a process in which a plasma jet is applied to the aforementioned water repellent film to dissipate the water repellency and (2) a process where a water repellent solution is applied to the area without the water repellent function.

[Claim 2] In the repair treatment method of water repellent film described in claim 1, the aforementioned plasma irradiation is applied and the substrate is cooled and the aforementioned glass surface is activated.

[Claim 3] In the repair treatment method of water repellent film described in claim 1, the aforementioned water repellent film is a water repellent film having a water repellent layer on a silica type substrate layer.

[Claim 4] In the repair treatment method of water repellent film described in claim 1, the aforementioned water repellent film is a water repellent film having a water repellent layer formed integrally with the silica type substrate layer.

[Claim 5] In the repair treatment method of water repellent film described in claim 1, the conditions of the aforementioned plasma irradiation are set as shown below.

Distance from lighting nozzle to substrate: 5 to 15 mm

Mobility of lighting nozzle parallel to the glass substrate surface: 1 to 60 mm/sec·time

[Claim 6] A repair treatment method characterized by the fact that the treatment includes (1) a process where a plasma is applied to the surface of a glass substrate and cleaning and activation of the surface of the aforementioned glass substrate is achieved, and (2) a process where the glass substrate surface with the plasma applied is coated with a water repellent solution.

[Detailed description of the invention]

[0001]

[Technical field of the invention] The present invention pertains to a repair treatment method of water repellent film used for automobile glass, etc. The invention further pertains to a repair treatment method of glass surfaces.

[0002]

[Prior art] In order to provide water repellency to automobile glass, it is necessary to achieve firm bonding of the glass surface and the water repellent film to increase durability of the water repellency.

[0003] As methods used in the past, (1) cleaning of the glass surface shortly before application of water repellency, (2) providing a substrate layer, etc. can be mentioned.

[0004] As specific cleaning methods used in (1) above,

1. cleaning with alcohol,

2. cleaning with an abrasive,

3. cleaning with ultraviolet light, etc. can be mentioned.

[0005] Furthermore, when the above-mentioned method (2) is used and a substrate layer is provided, the bonding strength of the glass surface and water repellent film can be increased and prevention of alkali diffusion from the glass can be achieved, and as a result, deterioration in the water repellent film can be prevented.

[0006] However, even when the bonding strength between the glass surface (substrate layer) and water repellent film is increased and durability of the water repellent film is increased, deterioration in the water repellency is reduced within several years under the harsh

environments of automobile usage; thus, repair treatment is required to maintain the water repellent effect.

[0007]

[Problems to be solved by the invention] In the above-mentioned repair treatment, it is desirable when the existing water repellent film that has deteriorated over time is removed since activation of the surface of the glass (substrate layer) is otherwise inhibited for application of the new water repellent film.

[0008] On the other hand, in order to increase bonding between the glass surface and water repellent film, or to increase durability, it is desirable when the above-mentioned substrate layer is provided.

[0009] However, the repair treatment is a treatment with the premise of after market installation, and in the above-mentioned cleaning method, removal of the water repellent film alone is not possible without removing the substrate layer as well.

[0010] Furthermore, regardless of the repair treatment, a clean surface is required for formation of a new water repellent film on the surface of the glass so as to increase the bonding strength between the glass surface and the water repellent film. Furthermore, as in the case of the repair treatment, a cleaning treatment is a prerequisite of after market installation, and the problems described below may occur when the above-mentioned cleaning is done.

[0011] 1. Cleaning with alcohol is the most convenient cleaning method, and there is no adverse effect on the substrate layer; thus, application of the method is possible in after market installation. Furthermore, thorough cleaning can be achieved with regard to soil based on organic components with low adhesion such as the oily film adsorbed. However, adequate cleaning for removal of the deteriorated water repellent film is not possible.

[0012] 2. Adequate cleaning can be achieved with regard to soil based on organic components such as oily film and soil based on inorganic components such as water marks when cleaning is done with an abrasive. However, the cleaning power is based on the polishing strength of the

abrasive; thus, cleaning power is too intensive and the thin substrate layer is polished as well. Thus, when the repair treatment is applied directly right after cleaning by means of polishing, water repellency of the water repellent film becomes inadequate at times since the effect of the substrate layer is lost.

[0013] Furthermore, it is necessary to perform the above-mentioned process with water, and disposal of the waste water containing the abrasive is required after cleaning. Furthermore, adhesion of the abrasive deposit to the body may occur and damage to the molding around the glass may occur; thus, masking around the glass is required as well, and the process requires additional time. Furthermore, when excessive polishing is done, it may cause external flaws such as distortion of the glass.

[0014]

3. For cleaning by ultraviolet light, a water repellent film removal method based on UV/ozone treatment is disclosed in Japanese Kokai [Unexamined] Patent Application No. Hei 9-142886. The substrate layer is not affected by the above-mentioned cleaning method but ozone is required for the treatment. Therefore, it is desirable when a facility for removal of ozone is provided.

[0015] Furthermore, due to low treatment efficiency, a treatment time of at least 10 minutes is required in the above-mentioned method. In addition, large equipment is required and treatment for the glass of automobiles is difficult, and application in the after market poses a problem.

[0016] Based on the above background, the purpose of the present invention is to provide a simple treatment method with improved bonding between the glass surface and water repellent film in repair treatment of the water repellent film used for automobiles, etc.

[0017] In specific terms, a method in which a repair treatment method for water repellent film is provided by dissipating the water repellency of the water repellent film without losing the function of the substrate layer, in a water repellent film having a substrate layer, while keeping the substrate layer intact.

[0018] Needless to say, the treatment of the present invention may be used for the repair

treatment of water repellent film without a substrate layer as well. Furthermore, the method may be used as a cleaning method at the time of formation of a new water repellent film.

[0019] In the specification of the present invention, the term "cleaning" includes the case of a treatment for achieving initial surface activity of the substrate layer as well as cleaning the surface of the substrate.

[0020]

[Means to solve the problem] In order to eliminate the above-mentioned existing problems, the point of the present invention is removal of the water repellent film and surface activation and cleaning of the substrate are done first by a plasma jet; then, the repair treatment is applied.

[0021] In other words, Claim 1 of the present invention is that a repair treatment method for water repellent film having a substrate layer characterized by the fact that the treatment includes (1) a process in which a plasma jet is applied to the aforementioned water repellent film to dissipate water repellency and (2) a process where a water repellent solution is applied to the area without the water repellent function.

[0022] Claim 2 of the present invention is that the aforementioned plasma irradiation is applied and the substrate is cooled and the surface of the aforementioned glass substrate is activated in the repair treatment method of water repellent film described in Claim 1.

[0023] Claim 3 of the present invention is that the aforementioned water repellent film is a water repellent film having a water repellent layer on a silica type substrate layer in the repair treatment method of a water repellent film described in Claim 1.

[0024] Claim 4 of the present invention is that the aforementioned water repellent film is a water repellent film having a water repellent layer formed integrally with the silica type layer in the repair treatment method of water repellent film described in Claim 1.

[0025] Claim 5 of the present invention is that the irradiation conditions of the aforementioned plasma are set as shown below in the repair treatment method of water repellent film described in Claim 1.

Distance from lighting nozzle to substrate: 5 to 15 mm

Mobility of lighting nozzle parallel to the glass substrate surface: 1 to 60 mm/sec·time

[0026] Claim 6 of the present invention is that the treatment includes (1) a process where a plasma is applied to the surface of the glass substrate and cleaning and activation of the surface of the aforementioned glass substrate is achieved, and (2) a process where the glass substrate surface with the plasma applied is coated with a water repellent solution in the repair treatment method.

[0027] According to the method of the present invention, removal of the substrate layer does not occur at the time of removal of the water repellent film and the substrate layer is left intact. Therefore, firm bonding between the glass surface and water repellent film can be achieved at the time of the water repellency treatment, and furthermore, durability of the water repellent film is increased as a result of prevention of alkali diffusion from the glass.

[0028] The painted surfaces of automobiles, etc. are not adversely influenced; thus, masking of areas other than the treated surface is not required.

[0029] Furthermore, with the device used in the method of the present invention, it is possible to take application to the after market into account. In other words, it is possible to produce a compact treatment device for removal of the water repellency film, and furthermore, wheels may be attached to the power supply to make the device portable.

[0030] Furthermore, a compact head member of the device that generates the plasma jet may be used so that handling with one hand is possible. For the head member, only a cable for supplying high voltage from the power supply to be applied between the electrode rod and electrode plate, and a power supply cable to air flow generator are connected to the head member; thus, handling is easy.

[0031] Therefore, the treatment can be easily applied to glass mounted in an automobile; thus, the treatment can be easily applied in after market applications.

[0032]

[Embodiment of the invention] First, the generator used for the plasma jet is explained below. Fig. 1 is a schematic view of plasma jet generator. The above-mentioned generator comprises power supply 50 in which supplies power to generate the plasma jet at light nozzle 10 and cart 51 and cables 60 that connect these. Though not shown in the figure, the high voltage supply cable for feeding high voltage from power supply 50 is connected to the electrode inlet of the light nozzle 10 and the electrode plate, and furthermore, power supply cable is connected to the motor of the air flow generator.

[0034] Power supply 50 of the plasma jet generator is mounted on cart 51 having wheels 52, leg 53, and handle 54. Furthermore, the plasma jet generator shown in the figure is an example where two light nozzles 10 are provided. The size of the power supply 50 is 508 x 229 x 610 mm, and weight if 58 Kg. The above-mentioned plasma jet generator is compact and equipped with wheels; thus, transporting of the power supply 50 by the worker is made easy.

[0035] Furthermore, main power supply switch 55, two light nozzles and corresponding operating lamps 56, On switch 57 and Off switch 58 are provided.

[0036] Fig. 2 is an enlarged front view of the plasma jet light nozzle 10 used in the present invention, and shows the condition where the plasma jet is applied to the object (water repellency glass plate 40).

[0037] Plasma jet light nozzle 10 has a structure where blower mount member 13 is attached to the main unit 12 provided with a flow path for the air flow generated by a motorized fan, etc.

[0038] Fig. 3 is a cross-section view at 2-2 of Fig. 2. In the figure, motor 21 and fan 23 attached to the motor shaft are provided on blower mount member 13. Furthermore, swirl chamber 24 that surrounding the above-mentioned fan 23 and air inlet port 25 provided with mesh 26 are formed on blower mount member 13 and function as the overall air flow generator 20.

[0039] Furthermore, electrode rod 30 is inserted from the upper part of the main unit 12. Furthermore, electrode plate 31 is provided at the lower part of the main unit 12 facing the aforementioned flow path 11.

[0040] When high voltage is applied to the space between electrode rod 30 and electrode plate 31, arc discharge 32 is generated between electrode rod 30 and electrode plate 31 and plasma is generated. At this time, high-speed air flow is supplied to the above-mentioned space from the aforementioned air flow generator 20. In this case, high-speed plasma jet 33 is emitted from the nozzle 14 and reaches the object (for example, water repellency glass plate 40).

[0041] The feature of the plasma jet treatment used in the present invention is explained below.

A plasma generated in the air is utilized; thus, it is not necessary to provide a booth, etc. for the treatment.

A treatment without thermal influence on the treated object is made possible.

Neither a cleaning process nor a drying process is needed after the treatment, and a different treatment can be applied immediately.

A dry process is used.

Control and operation are easy compared to other surface treatment methods.

[0042] Therefore, the method is effective when used as a pre-treatment for adhesion, bonding, coating, printing, etc. Furthermore, as substrates used in the present invention, plastics, metals, rubbers, glasses, ceramics, etc. can be mentioned.

[0043] Furthermore, for the mechanism of the plasma jet treatment, decomposition of the molecular bonds on the surface of the substrate by the plasma particles can be mentioned. For example, in a perfluoroalkylsilane that exhibits water repellency, decomposition of the alkyl group and Si bond occurs when a plasma jet treatment is applied, and as a result, water repellency disappears.

[0044] Furthermore, when soil (organic material) exists on the surface of the substrate, the soil is decomposed by the plasma particles; thus, the surface of the substrate is cleaned as well. [0045]

Furthermore, hydrophilic groups such as OH groups are formed on the treated surface that form the reaction activation points with the water repellent film, thus, surface activity can be increased. Therefore, when the water repellency treatment is applied after the above-mentioned

plasma jet treatment, bonding of the water repellent agent is increased and a water repellent film with high durability can be formed.

[0046] Furthermore, depending on the substrate used, formation of molecular level pattern on the surface of the substrate can be expected based on the plasma particles. In this case, an anchoring effect for the process that follows can be expected as well.

[0047] The processing capacity of the plasma jet is determined by the distance between the head and the work, head feed speed and number of treatments when the output is constant.

[0048] For treatment conditions used in the present invention, it is desirable when the distance between the head and the work is in the range of 5 to 15 mm when the output of the plasma jet light is 0.5 kW per nozzle. When the above-mentioned distance is less than 5 mm, the treated surface may be thermally affected. On the other hand, when the distance exceeds 15 mm, processing efficiency is reduced since it is outside the range of effective plasma jet treatment.

[0049] Furthermore, head feed rate converted to a per-treatment time of 100 mm/(sec·time) or below is desirable, and 60 mm/(sec·time) or below is further desirable, and 20 mm/(sec·time) or below is especially desirable.

[0050] In this case, when the plasma jet treatment is done many times for the same treatment surface, the head feed rate converted to per-treatment can be obtained when actual head feed rate is divided by the number of treatments performed.

[0051] Furthermore, when the head feed rate exceeds 100 mm/(sec·time), treatment performance likely to be insufficient, and full removal of the water repellency may not be possible. On the other hand, when the head feed rate is too slow, covering of an adequate treatment area is not possible and it is not cost effective. Therefore, the treatment is applied at a feed rate for which the treatment capacity is reduced.

[0052] Furthermore, lighting output of the plasma jet can be regulated by controlling the frequency and voltage applied by the power supply unit. And the above-mentioned output is in the range of 0.1 to 1.0 kW per nozzle. Furthermore, light output of the plasma jet may be

selected in a stepwise manner according to the material of the substrate to be treated or the water repellent film formed.

[0053]

[Working Examples]

The present invention is explained in further detail with working examples below.

[0054]

(Production of a water repellent film with substrate) Stirring was provided for 98.6 g of ethanol (product of Nakarai-Tex Co., Ltd.) with 0.4 g of tetraethoxysilane (product of Shinetsu Silicone Co., Ltd.) and 1 g of concentrated hydrochloric acid (35 wt%, product of Kanto Chemical Co., Ltd.) and a treatment solution for a silica substrate layer was produced.

[0055] Furthermore, a cleaned soda-lime silicon glass substrate (300 x 300 mm) was coated with the above-mentioned treatment solution for silica substrate layer at room temperature and under humidity of 30% RH using the flow coating method, drying was done for approximately 1 minute to form a silica substrate layer with a thickness of approximately 40 nm on the glass substrate.

[0056] When the hardness of the above-mentioned silica film was measured using a pencil hardness test, damage did not occur to the film even when scratches were formed with an H pencil.

[0057] Subsequently, 1 g of $CF_3(CF_2)_7(CH_2)_2Si(OCH_3)_3$ (heptadecafluorodecyltrimethoxysilane, product of Toshiba Silicone Co., Ltd.) was dissolved in 98 g of ethanol to produce a water repellent treatment solution (repair treatment solution); then, 1 g of 0.1 N hydrochloric acid was added, and the solution was stirred for 1 hour to produce a water repellent treatment solution (repair treatment solution).

[0058] Furthermore, the surface of a glass substrate coated with a silica substrate layer was coated with a cotton cloth soaked with 3 mL of the above-mentioned water repellent treatment solution; excess water repellent treatment solution was wiped off with a fresh cotton cloth to

provide a water repellent treated glass.

[0059] (Production of water repellent film without a substrate layer) A cleaned surface of a soda-lime silicon glass substrate (300 x 300 mm) was coated with a cotton cloth soaked with 3 mL of the above-mentioned water repellent treatment solution; excess water repellent treatment solution was wiped off with a fresh cotton cloth to provide a water repellent treated glass.

[0060] The above-mentioned two types of water repellent glasses, one with a substrate layer and one without a substrate layer, were stored outside for the same length of time and a repair treatment was done for samples in which the contact angle had become 70 degrees or below.

[0061] First, for the above-mentioned two different types of glass, three different treatments, 1. plasma jet treatment, 2. celico [transliteration] cleaning treatment, and 3. alcohol cleaning treatment, were applied, and with repeated treatment production of a water repellent, treated glass was achieved. In this case, samples with a substrate layer were 1-1, 1-2 and 1-3, and samples without a substrate layer were 2-1, 2-2, and 2-3.

[0062] In this case, the plasma jet treatment was applied according to the conditions shown below.

Treatment apparatus: plasma jet treatment apparatus (PJ-1), product of Corotec Co.

Treatment condition: output 0.5 kW/nozzle

Distance between head and work: 8 mm

Conversion head feed rate: 20 mm/(sec·time)

plasma jet flow rate: approximately 7 m/sec

Furthermore, when purified water was dropped onto the surface of the glass after the above-mentioned treatment, uniform wetting was observed and dissipation of water repellency was confirmed.

[0063] In this case, celico [transliteration] cleaning was performed with a glass cleaning paste (Kirobin: product of Takehara Co., (Ltd.)) containing celico as thorough polishing of the glass surface was done, and subsequently the glass was rinsed clean with water. Furthermore, when

purified water was dropped onto the surface of the glass after the above-mentioned treatment, uniform wetting was observed and dissipation of water repellency was confirmed.

[0064] Furthermore, alcohol cleaning was provided as the surface of the glass was cleaned with a cotton cloth soaked with ethyl alcohol. Furthermore, when purified water was dropped onto the surface of the glass after the above-mentioned treatment, uniform wetting was not observed and removal of the water repellent film was inadequate.

[0065] For the above-mentioned water repellent treated glass, measurements were made of the initial static contact angle (hereinafter referred to as contact angle) as a 2-mg water droplet was applied to the treated glass surface using a contact angle meter (CA-DT, product of Kyowa Interface Science Co., Ltd.).

[0066] For the friction test, a dry cloth was attached to a reciprocating friction tester (product of Shinto Chemical Co., Ltd.) and rubbing was done for 3000 reciprocating motions on the surface of the water repellent film of the water repellent treated glass under a load of 0.3 kg/cm^2, and the contact angle was subsequently measured.

[0067] For the weather resistance test, UV tester (I-Super W-13, product of Iwasaki Electric Co., Ltd.) was used and ultraviolet was applied at an ultraviolet intensity of 76±2 mW/cm^2, black panel temperature of 48±2 deg C and continuous radiation of 600 hours, and the contact angle was subsequently measured.

[0068] Measurement results for the contact angle measured for each of the above test items are shown in Table I below. In this case, when the contact angle after the friction test and after the weather resistance test is 80° or higher in both cases, a "double circle" is used; if one or the other is 80° or above, a "circle" is used; and if both contact angles are below 80°, an "X" is used.

[0069] [Table I]

Sample	Form of hydrophilic film	Method of removing hydrophilic film	Contact angle after abrasion test (°)	Contact angle after weather resistance test (°)	Evaluation according to criteria
1-1	Substrate layer	plasma J	100	85	"double circle"
1-2	Substrate layer	celico cleaning	85	75	"circle"
1-3	Substrate layer	alcohol	69	59	X
2-1	No substrate layer	plasma J	83	72	"circle"
2-2	No substrate layer	celico cleaning	85	75	"circle"
2-3	No substrate layer	alcohol	61	57	X

[0070] As shown in the results obtained above, removal of water repellent film is possible while keeping the substrate layer intact when plasma jet treatment is applied to a water repellency glass having a substrate layer. Furthermore, increase in bonding between the substrate layer and water repellent film is made possible when a water repellent solution is further applied. And furthermore, superior durability of the water repellent film can be achieved when plasma jet treatment is applied to a glass without a substrate layer rather than alcohol cleaning.

[0071] In this case, the water repellent film of sample 1-1 is a water repellent film having a two-layer structure having a substrate layer. The plasma jet treatment used in the present invention can be used effectively for a water repellent film having a two-layer structure as well as for a water repellent film having a water repellency layer formed integrally with the silica type

skeleton layer.

[0072] In the above-mentioned integral water repellent film, perfluoroalkyl groups having water repellency are naturally arranged on the surface. And in this case, water repellency can be removed upon application of a plasma jet.

[0073] As a method of manufacturing an integral form water repellent film, the method disclosed in Japanese Kokai [Unexamined] Patent Application No. Hei 11-71682 can be mentioned. A plasma jet is applied to the above-mentioned integral water repellent film produced by the above-mentioned method. When purified water was dropped onto the surface, uniform wetting was observed and dissipation of water repellency was confirmed.

[0074] Furthermore, when the above-mentioned plasma jet treatment was used for cleaning when a water repellent treatment is applied directly to the glass substrate, water repellency performance greater than that of sample 2-1 was achieved. This indicates that the plasma jet can be used effectively as a cleaning method when a water repellency treatment is applied directly to a glass substrate.

[0075]

[Effect of the invention] According to the repair treatment method for water repellent films of the present invention, the effects described below can be achieved.

[0076] Repair treatment is made possible while maintaining the effect of the substrate layer since adverse influence on substrate layer is absent when a plasma jet treatment is used.

The plasma jet treatment is effective for cleaning and activation of the substrate surface, and can be used as a pre-treatment as well.

The head member of the plasma jet apparatus is easy-to-handle, and the treatment itself is a dry process; thus, treatment of the glass is possible while it is mounted on automobiles without having an adverse effect on the paint, etc., surrounding the glass.

The above-mentioned plasma jet apparatus is compact and equipped with wheels; thus, transporting is easy and after market application is possible.

[Brief description of the figures]

[Fig. 1] A schematic view of the plasma jet generator.

[Fig. 2] A front view of the plasma jet lighting nozzle used in the present invention.

[Fig. 3] Cross-section view of the plasma jet lighting nozzle.

[Explanation of codes]

1: Plasma jet generator

10: Plasma jet lighting nozzle

11: Flow path

12: Main unit

13: Blower mount section

14: Nozzle

20: Air stream generating area

21: Motor

22: Motor shaft

23: Fan

24: Swirl chamber

25: Air intake

26: Net

30: Electrode rod

31: Electrode plate

32: Arc discharge

33: Plasma jet

40: Water repellent glass sheet

50: Power supply

51: Carrier

52: Wheels

53: Leg

54: Handle

55: Main powder supply switch

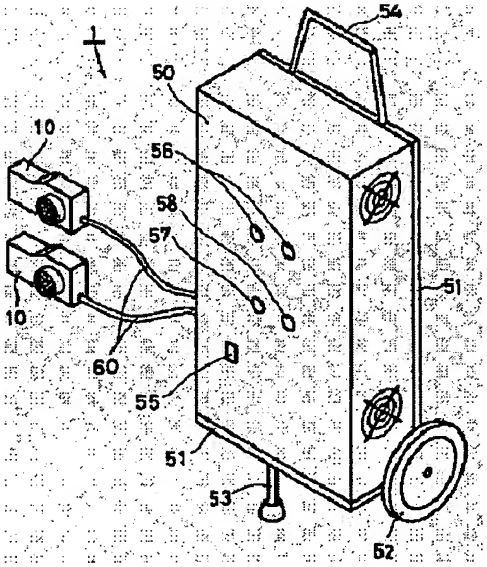
56: Operation lamp

57: ON switch

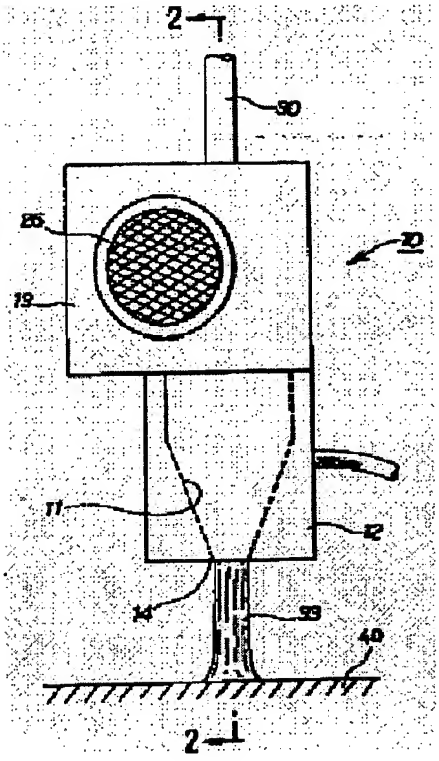
58: OFF switch

60: Cable

[Fig. 1]



[Fig. 2]



[Fig. 3]